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NINTH QUARTERLY PROGRESS REPORT
Covering the Period October 1, 1977, to December 31, 1977

on

**EVALUATION OF SELECTED CHEMICAL PROCESSES
FOR PRODUCTION OF LOW-COST SILICON**
(Phase II)

JPL Contract 954339

Silicon Material Task
Low-Cost Silicon Solar Array Project

to

JEP PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY

by

J. M. Blocher, Jr., and M. F. Browning

January 20, 1978

This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, under NASA Contract NAS7-100 for the U.S. Department of Energy, Division of Solar Energy.

The JPL Low-Cost Silicon Solar Array Project is funded by DOE and forms part of the DOE Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays.

(NASA-CR-157073) EVALUATION OF SELECTED
CHEMICAL PROCESSES FOR PRODUCTION OF
LOW-COST SILICON PHASE 2. SILICON MATERIAL
TASK, LOW-COST SILICON SOLAR ARRAY PROJECT
Quarterly Progress (Battelle Columbus Labs., G3/44

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BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

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ERRATA

On page 2, paragraph 1, line 6 of the Eighth Quarterly Report (ERDA/JPL 954339-77/8), the word "concentration" should read "condensation".

NINTH QUARTERLY PROGRESS REPORT
Covering the Period October 1, 1977, to December 31, 1977

on

EVALUATION OF SELECTED CHEMICAL PROCESSES
FOR PRODUCTION OF LOW-COST SILICON

JPL Contract 954339

Silicon Material Task
Low-Cost Silicon Solar Array Project

to

JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY

from

BATTELLE
Columbus Laboratories

January 20, 1978

ABSTRACT

Progress from October 1, 1977, through December 31, 1977, is reported in the design of the 50 MT/year experimental facility for the preparation of high-purity silicon by the zinc vapor reduction of silicon tetrachloride in a fluidized bed of seed particles to form a free-flowing granular product.

Progress was made in all aspects of the design of the 50 MT/year experimental facility. Design of the silicon tetrachloride purification facility by Pace Engineers, Inc., of Houston, Texas, is nearing completion and is expected to be concluded during January, 1978. Progress was made in the design of the Zinc/ ZnCl_2 by-product condenser, and in the more conventional items of the facility by Raphael Katzen Associates, International, Inc., of Cincinnati, Ohio. Progress was also made at Battelle in the

non-conventional items, the fluidized bed, zinc feed system, zinc vaporizer, and electrolytic cell. Design decisions have been made which leave unresolved questions only in the case of the zinc pump, zinc vaporizer, and electrolytic cell. These are expected to be resolved early in the next quarter, so that the design for cost estimation purposes can be concluded in February.

Calculations and experimental work have defined the limitations of a graphite tray-type vaporizer for zinc, and the alternative of directly coupling induction heat to zinc is being explored.

Problems with the hot-wall graphite-lined electrolytic cell have led to adoption of cold-wall design (contained insulation) for the experimental facility; however, it is concluded that most of the problems stemmed from excessive moisture in the ZnCl_2 used to form the synthetic ZnCl_2/KCl mixtures, and that these would be greatly reduced with the anhydrous by-product of the experimental facility.

A total of 1.5 kg of quality-evaluation material has been prepared with semiconductor-grade seed during this report period and shipped to JPL. The concept of chlorination of residual silicon deposit on the fluidized-bed reactor wall was demonstrated as was the semicontinuous withdrawal of product in the course of preparing this material.

Progress was made in experimental evaluation of design options for the liquid zinc displacement feed device and the zinc vaporizer.

Experiments were initiated in a full-scale mock-up of the fluidized-bed unit to observe the effects of particle size, bed height, and relative flow through the gas distributor on fluidization, and on mixing of the two gas streams.

Materials compatibility tests showed that stainless steel was not attacked at 500 C by zinc chloride, but that zinc vapor penetrated the pores of ATJ graphite to attack the surrounding stainless steel at 850 C, thus confirming the necessity of rendering the graphite impervious for that type of construction.

INTRODUCTION

This is the Ninth Quarterly Progress Report covering the work at Battelle's Columbus Laboratories for DOE-JPL on the Evaluation of Selected Processes for the Production of Low-Cost Silicon.

This work is currently in the second phase, that of designing a 50 MT/year experimental facility for the preparation of silicon by the zinc vapor reduction of silicon tetrachloride on a fluidized bed of seed particles.

The Fifth/Sixth Quarterly Report of this series (ERDA/JPL 954339-77/5,6, April 29, 1977) summarizes the prior work and plans for the current program through design (Phase II), construction (Phase III), shakedown (Phase IV), and experimental operation (Phase V) of the experimental facility. The target date for completion of the design is February 15, 1978.

In writing this report, it is assumed that the reader will have the background of the Fifth/Sixth, Seventh, and Eighth Quarterly Reports. Accordingly, progress and status changes since the Eighth Quarterly Report are reported with a minimum of background, in two sections, Design of the Experimental Facility, and Experimental Support Programs.

DESIGN OF THE EXPERIMENTAL FACILITY

The basic process flow diagram and mass and energy flow sheets for the 50 MT/year experimental facility were presented as Figure 1 and Tables 1 and 2 of the Eighth Quarterly Report (ERDA/JPL 965339-77/8, October 20, 1977). Included also as Figure 2 of that report was a preliminary plant layout, drawn to give assurance that the experimental facility would fit into an available building.

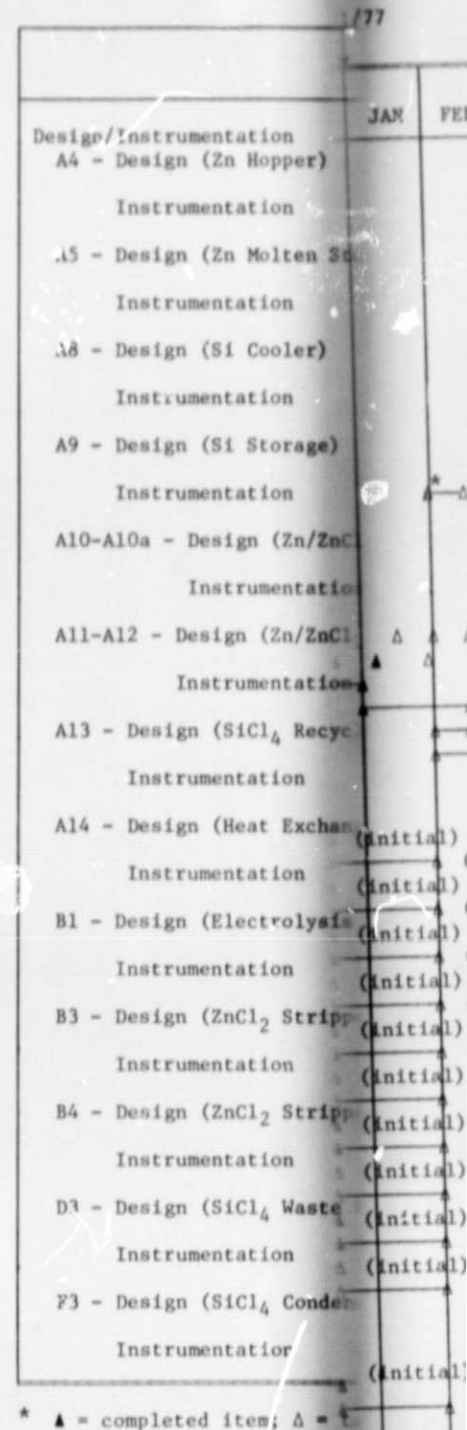
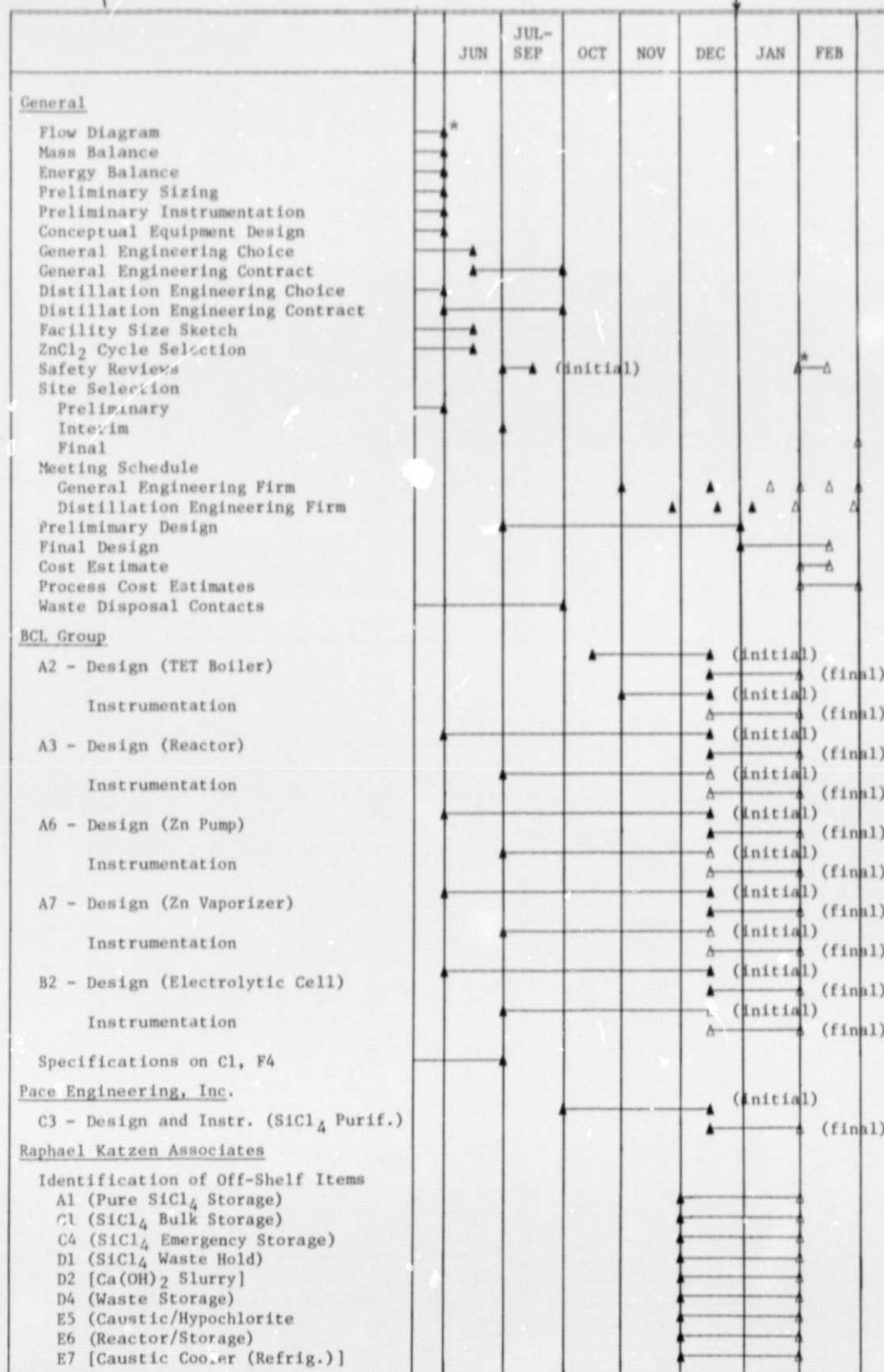
During the quarter covered by this report, intensive activity has been maintained in the area of plant design in cooperation with Raphael Katzen Associates, International, Inc. (RKAII), of Cincinnati, Ohio, and with Pace Engineers, Inc. (Pace), of Houston, Texas. Pace has the task of designing

the silicon tetrachloride purification system, while RKAI is handling the design of the more conventional items other than SiCl_4 purification, and cooperating with Battelle's Columbus Laboratories (BCL) on the less conventional items. The BCL design responsibilities include the less conventional items and oversight of the entire design effort. Figure 1 shows the current design schedule. This schedule gives a fairly complete picture of the progress of the design effort. However, it should be noted that in the course of interaction with RKAI, changes in design have been proposed which may alter the function and therefore the identity of certain items given in Figure 1. As these changes are tentative, it is believed desirable at this point to retain the item identity given previously in this figure (as Figure 7 of the Eighth Quarterly Report). It will be noted that Equipment Item C2 (SiCl_4 Reserve) has been deleted and C4 (SiCl_4 Emergency Storage) has been added.

Because of the state of flux of the design of the experimental facility, it is not practical to recount a full description of the design as of a given date (e.g., at end of quarter, December 31, 1977). Rather, a general statement of progress will be made, coupled with the presentation of selected figures which will be representative of that progress, but are not expected to be understood in detail by other than those who are intimately involved. Such detailed presentation will be reserved for the final report.

RKAI Progress

Figure 2 is a preliminary flow diagram of the reaction section of the facility, drawn up for critique at a meeting on December 15, 1977, at BCL. The major additions, in principle, to earlier flow diagrams is the provision of a heat exchanger system for the zinc/ ZnCl_2 condenser, A10, involving a high-temperature heat exchange fluid "Therminol" whose heat content is finally dissipated by cooling in Units A17 and A18 of the diagram. A significant change made as the result of subsequent discussions has been the combination of A4 with A4' and A5 with A5' into single units of 50 MT/year capacity to feed both of the 25 MT/year fluidized-bed system branches.



* ▲ = completed item; Δ = in progress

FIGURE 1. DESIGN SCHEDULE, EXPERIMENTAL FACILITY (1/20/78)

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	JUN	JUL-SEP	OCT	NOV	DEC	JAN	FEB
Design/Instrumentation							
A4 - Design (Zn Hopper)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
A5 - Design (Zn Molten Storage)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
A8 - Design (Si Cooler)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
A9 - Design (Si Storage)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
A10-A10a - Design (Zn/ZnCl ₂ Condenser)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
A11-A12 - Design (Zn/ZnCl ₂ Stripper)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
A13 - Design (SiCl ₄ Recycle Condens.)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
A14 - Design (Heat Exchanger)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
B1 - Design (Electrolysis Feed)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
B3 - Design (ZnCl ₂ Stripper)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
B4 - Design (ZnCl ₂ Stripper)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
D3 - Design (SiCl ₄ Waste Process)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)
F3 - Design (SiCl ₄ Condenser)					▲ (initial)		▲ (final)
Instrumentation					▲ (initial)		▲ (final)

* ▲ = completed item; Δ = target item.

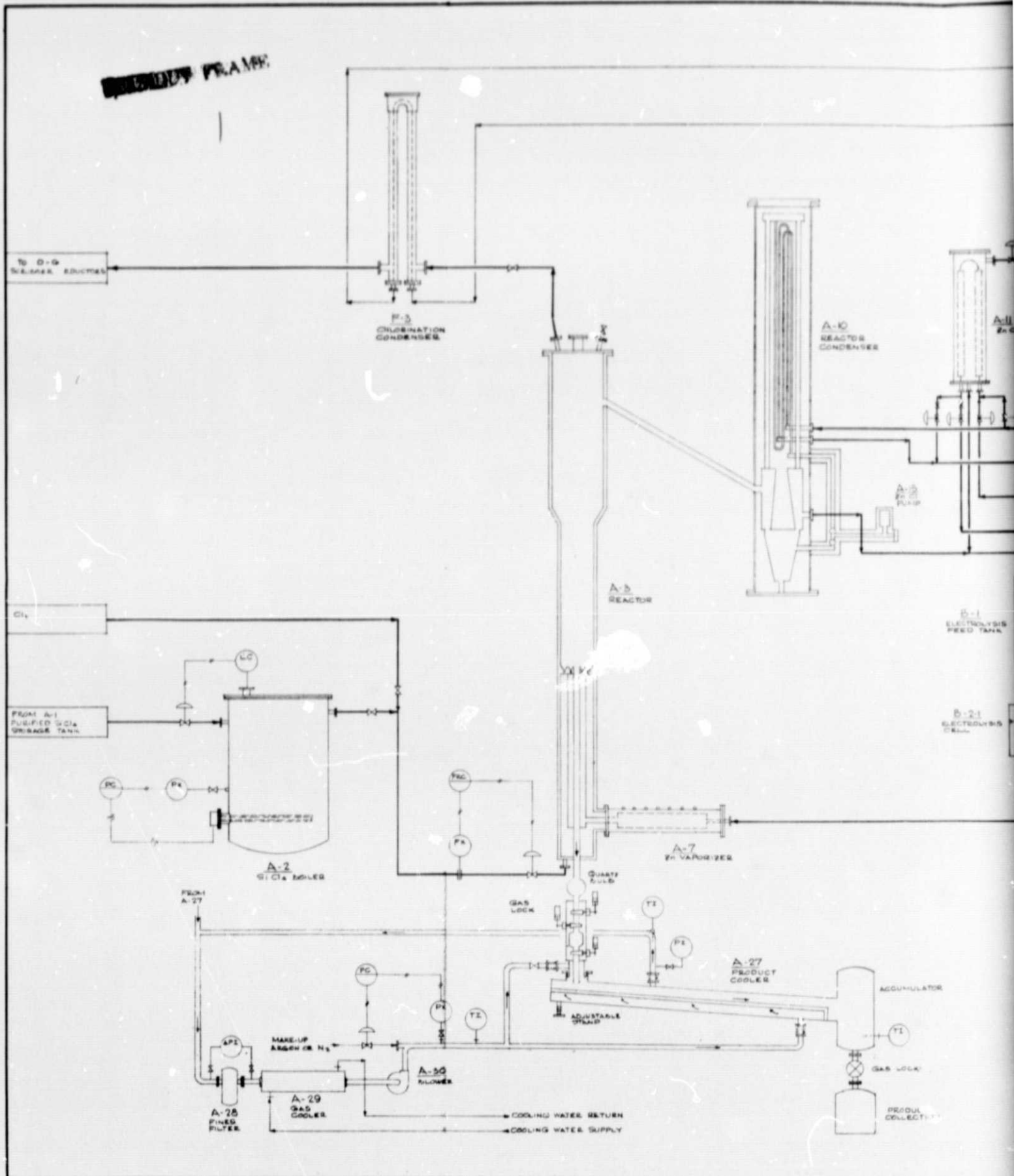
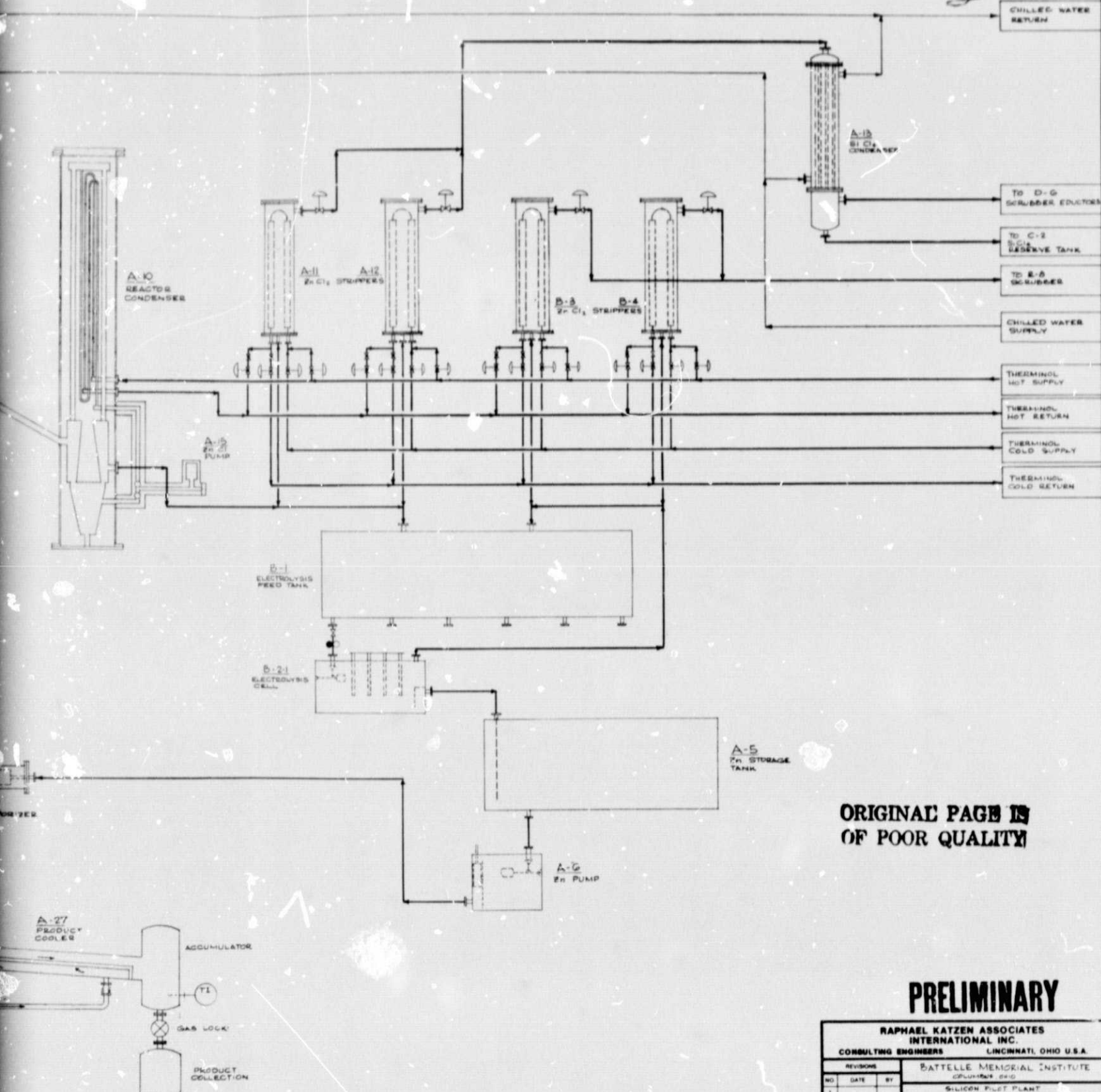


FIGURE 2. PRELIMINARY FLOW DIAGRAM, REACTOR SECTION
(RKAII DRAWING NO. WH 249-02)



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PRELIMINARY

REVISIONS			RAPHAE KATZEN ASSOCIATES INTERNATIONAL INC. CONSULTING ENGINEERS LINCINNATI, OHIO U.S.A.	
NO.	DATE	BY	BATELLE MEMORIAL INSTITUTE COLUMBUS, OHIO	
1			SILICON FLUORIDE PLANT	
2			FLOW SHEET	
3			REACTION & RECOVERY SYSTEM	
4			DRAWN BY: SWL	DATE: 12-24-1978
5			CHECK BY: SCALE	DRAWING NO: WH 249-02

Similarly, the electrolysis feed tanks, B1 and B1', have since been combined, so that in essence, the six 5000-ampere electrolytic cells can be fed with the product of either of the zinc/ZnCl₂ condensers, A10 and A10', and can be used in any combination to recycle zinc to either of the 25 MT/year branches.

Zinc/ZnCl₂ Condenser

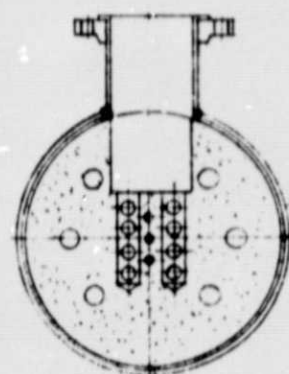
The zinc/ZnCl₂ condensers, A10 and A10', are designed to be of the wet-wall type with ZnCl₂ as the recirculated fluid. This design principle is warranted by the nature of the condensate, containing silicon "dust", which, if allowed to impinge on a dry surface, would tend toward plugging, depending upon particle size, shape, and associated entrainment characteristics. It should be noted that as the temperature of the by-product gas is decreased and the zinc and ZnCl₂ condense out, the gas velocity for entrainment of silicon "dust" is ultimately decreased by a factor of 11.

Figure 3 shows a preliminary design of the wetted-wall zinc/ZnCl₂ condenser. A description (preliminary) of its operation follows:*

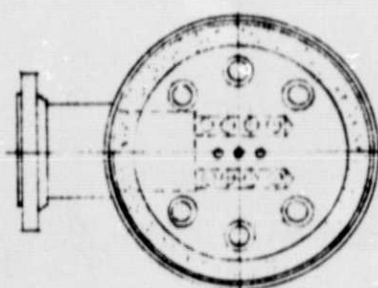
The cooling of the reactor off-gases can be divided into three segments -- condensation of the zinc, condensation of the zinc chloride, and gas cooling. Zinc condensation occurs essentially between 1475 F (802 C) and 1275 F (690 C), zinc chloride condensation from 1275 F (690 C) to about 1050 F (565 C), and the remainder of the cooling is essentially gas cooling. The condensation of zinc and zinc chloride can be efficiently handled in the A10 condenser. However, the gas cooling portion, below 1050 F (565 C), should be carried out in exchangers with a high surface per equipment volume in order to avoid unduly large equipment.

* Extracted from RKAI Communication, WH 249, December 2, 1977.

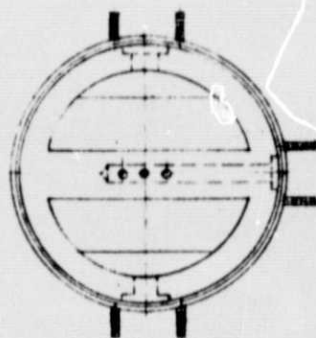
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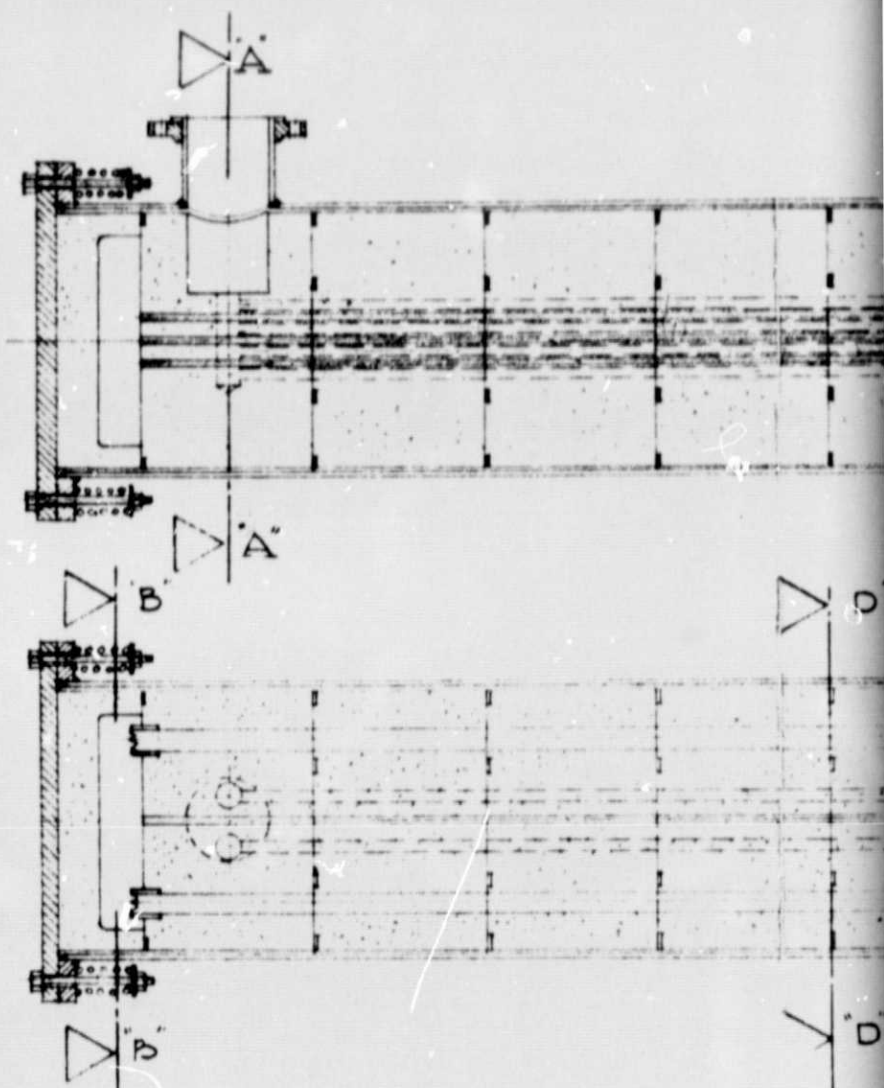
SECTION 'A-A'



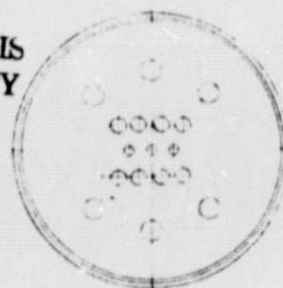
SECTION 'B-B'



SECTION 'C-C'



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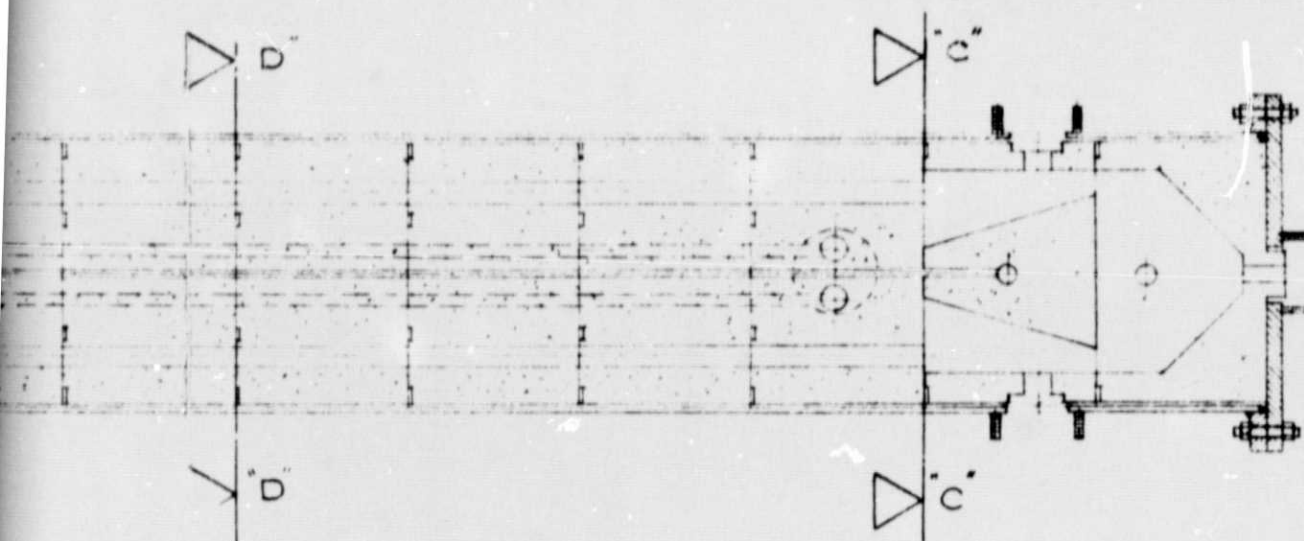
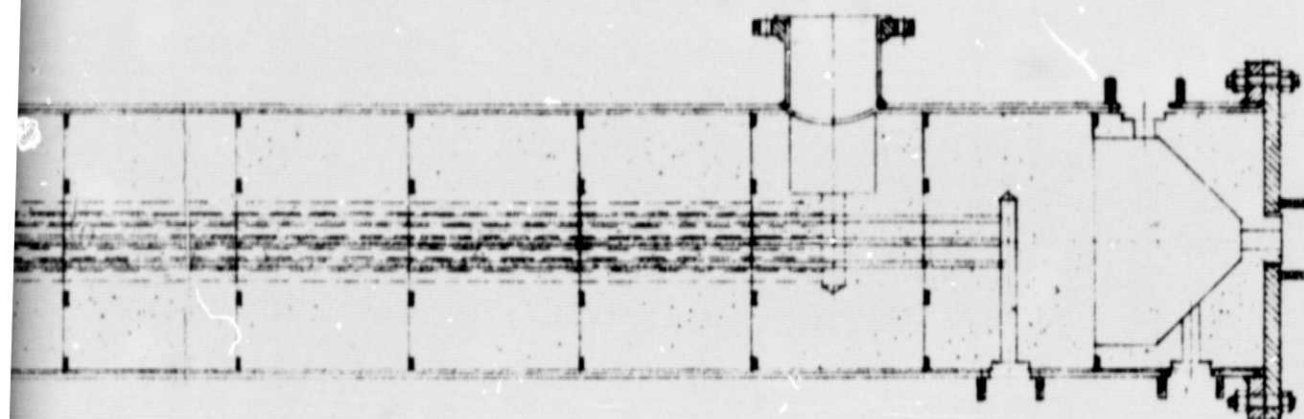
SECTION 'D-D'

PRELIM

FIGURE 3. PRELIMINARY DESIGN OF ZINC/ ZnCl_2 CONDENSER
(A10) (RKAI1 DRAWING NO. WH 249-05)

REPORT TEAM

2



PRELIMINARY

RAPHAEL KATZEN ASSOCIATES				
CONSULTING ENGINEERS		CINCINNATI, OHIO		
REVISIONS			BATTELLE MEMORIAL INSTITUTE COLUMBUS, OHIO SILICON PILOT PLANT ITEM A10 REACTOR CONDENSER	
NO.	DATE	BY		
1				
2				
3			DRAWN BY WWE DATE 1 DEC 1977	DRAWING NO. WH 249-05
4			CHKD BY SCALE 1" = 2"	

In the proposed configuration of Figure 3, reactor off-gas enters the bottom of the condenser and travels upward through the outer holes. If necessary, a two-pass arrangement can be used with the gas flowing back down through a second bank of holes to leave the exchanger at the bottom head. Zinc chloride is pumped to the top head and allowed to overflow the collars on each tube and down the tube walls. The zinc chloride circulation rate required to completely wet the tube wall is approximately 0.4 gpm per tube. This will insure that there can be no build-up of molten zinc and silicon dust on the tube walls and also allows the zinc chloride to serve as the cooling medium for the reactor off-gas. Zinc chloride will be supplied to the tubes at a temperature of 800 F (427 C), slightly above the melting point of zinc. Net condensate leaves the exchanger by overflow. The heat is removed from the zinc chloride by exchanging it against Therminol in the central portion of the exchanger. The zinc chloride circulating pump takes suction below the liquid level in the bottom of the exchanger and supplies zinc chloride to the three center tubes. The Therminol enters the top of the exchanger at approximately 700 F (371 C) and flows countercurrent to the rising zinc chloride. The Therminol leaving the bottom of the exchanger at about 730 F (388 C) is cooled in a conventional air- or water-cooled exchanger prior to being circulated back to the top of the unit.

Below a temperature of approximately 1050 F (565 C), the controlling heat transfer resistance is that of the gas film and the overall heat transfer coefficient is very low. For this reason, it is recommended that

cooling below 1050 F (565 C) be carried out in a shell and tube or block type exchanger with a high specific surface.

Preliminary calculations indicate that the zinc condensation can be carried out in three 1-1/2-inch-ID tubes with a length of approximately 5 feet. The bulk of the zinc chloride condensation can be carried out in a length of approximately 3 feet. Difficulty of heat transfer below 1100 F (593 C) is shown by the fact that it would require over 13 feet of the same tube to cool from the 1100 F (593 C) down to 932 F (500 C).

Refinement of the design of A10 and associated equipment is in progress.

Waste Treatment Section

On the basis of experience with similar systems, RKAI recommends that the SiCl_4 waste disposal system (D1-D4) and the chlorine-to-hypochlorite conversion system (E5-E7) be integrated with each other and with a scrubber system for handling vent products and the building air in case of leaks or spills. This has the advantage of using a single reactant, NaOH, for the two operations. The use of $\text{Ca}(\text{OH})_2$ for the SiCl_4 neutralization [to SiO_2 (hydrated)] was based on experience at Battelle and elsewhere indicating that plugging of the SiCl_4 vapor inlet in such systems is a problem with NaOH as the neutralizing solution. According to RKAI, this problem should be minimized by using an eductor arrangement through which relatively large volumes of NaOH solution are circulated. In recognition that the problem might be greatly reduced but not completely eliminated, spare eductors are provided for use when the constricted ones are being reconditioned. The construction is such as to simplify that operation. To confirm the feasibility of such an arrangement, a small eductor of the recommended design has been ordered for evaluation in the experimental support program.

A preliminary flow diagram of the waste treatment section is given in Figure 4.

A description of the proposed operation of the waste treatment section follows:*

Primary Silicon Tetrachloride Scrubber. The function of the primary silicon tetrachloride scrubber is to neutralize any waste streams that contain silicon tetrachloride. The neutralization of silicon tetrachloride vapors will be accomplished in Eductor D6. Since the eductor could ultimately become blocked, a spare is provided for this item. The liquid waste streams will be neutralized in the SiCl_4 scrubber tank D3. Neutralization of the silicon tetrachloride will be accomplished by using a sodium hydroxide solution which is maintained at a minimum of 2 percent sodium hydroxide (pH of 13.7). To remove the heat of neutralization, the recycle stream, which is used in the eductor, is cooled in a recycle cooler D5. Both the recycle cooler and the eductor have been sized to handle the peak silicon tetrachloride waste condition which would occur during the chlorination of the silicon reactor.

The net discharge from the primary silicon tetrachloride scrubber is discharged to the sewer. The net flow from the scrubber during normal operation (92 percent of operating time) is approximately 0.3 gpm. This amount should cause no environmental problems, especially since it will be diluted heavily (10 to 1) with cooling water coming from the recycle cooler. The gases from the primary scrubber are vented to the final vent gas scrubber.

* Extracted from RKAI Communication WH 249, December 2, 1977.

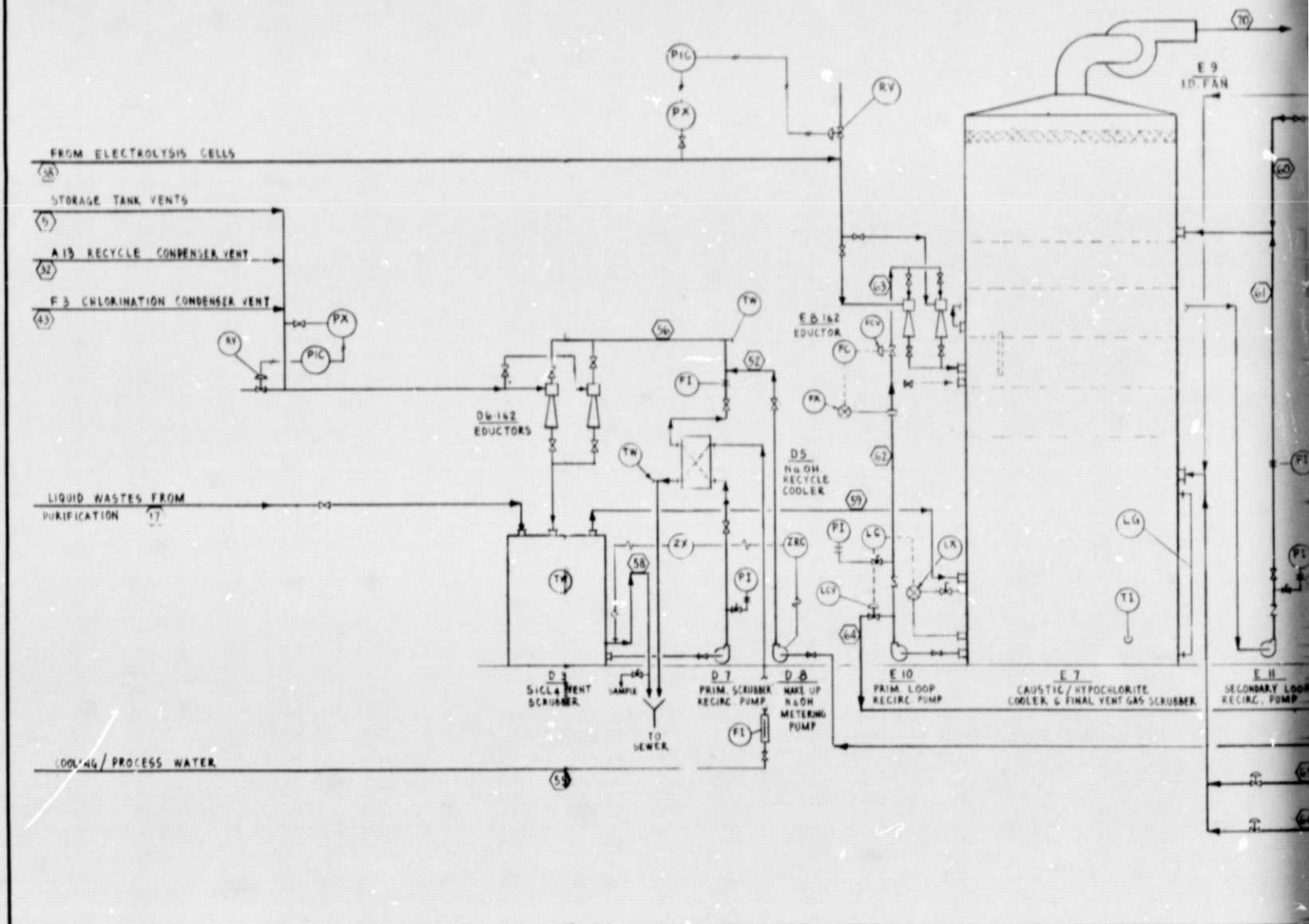


FIGURE 4. PRELIMINARY FLOW DIAGRAM, WASTE TREATMENT SECTION (RKAI1 DRAWING NO. WH 249-04)

DISCONTINUATION

2



REVISIONS			BATTELLE MEMORIAL INSTITUTE COLUMBUS, OHIO	
NO.	DATE	BY	SILICON PILOT PLANT	
1			WASTE TREATMENT SECTION	
2				
3				
4			DRAWN BY: D.C.P.	DATE: NOV. 30, 77
5			CHECKED BY: SCALE	DRAWING NO: WH 249-04

Final Vent Gas Scrubber and Hypochlorite/Caustic Cooler.

Chlorine from the electrolysis cells will be neutralized to hypochlorite in eductor E8. Since there is a trace amount of silicon tetrachloride in the chlorine stream which could lead to constriction, a spare has been provided for this eductor. Essentially all of the chlorine will react by the time it enters the vent scrubber E7. However, since there is a possibility of unreacted chlorine being discharged to the atmosphere, the scrubber consists of a two-loop system. The primary loop consists of recycling sodium hydroxide solution over five perforated trays. Air coming from area vents, outside air, and the vent from the primary SiCl_4 scrubber cools the reaction solution. The net hypochlorite solution is discharged from this primary loop into the hypochlorite storage tank E5. The secondary recirculation loop consists of two perforated trays. The net makeup of caustic solution, from caustic storage tank E6, is fed onto the top tray of this secondary loop. The net overflow from the secondary loop overflows into the primary loop, supplying the necessary caustic to neutralize the chlorine from the electrolysis cells. A demister is used to separate any entrained caustic solution. An ID fan is used to discharge the scrubbed air and inert gases to the atmosphere.

During the conference at BCL on December 15, 1977, refinements were suggested in the design which will be finalized by RKAI.

Pace Progress

Pace has been assigned the task of designing a silicon tetrachloride purification system to convert the commercially available material of the

purity indicated in Table 1 to one that will yield semiconductor-grade silicon, with the target composition given in the second column of Table 1. This is accomplished by a distillation system consisting of separate light-ends and heavy-ends columns with an intermediate surge tank. The light-ends and heavy-ends columns each contain 13.5 feet of packing equivalent for a total of 20 theoretical plates. The entire system is designed to operate as high as 60 psig if desired.

Figure 5, the process flow diagram, and Figure 6, an elevation drawing, are shown to give a perspective rather than to present the information in detail, such as will be done in the final report.

The purification facility is to be constructed skid mounted, and the skids and upper sections of the distillation column are to be assembled on location. The design task has proceeded rapidly, the items listed in Table 2 having been secured from Pace. Cost estimation and subsequent construction of this section of the 50 MT/year facility are facilitated by the fact that the components are of standard design or are available "off the shelf".

BCL Progress

Design work at BCL has progressed on the zinc pump, zinc vaporizer, fluidized-bed unit, and the electrolytic cell. In addition, contribution has been made to the design work carried out by RKAI and Pace.

Zinc Pump

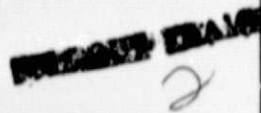
As noted in the Experimental Support section of this report, the basic design for the zinc pump, A6, appears to be feasible. Charges made as the result of experimental work will be discussed in that section of the report.

TABLE 1. SiCl_4 COMPOSITION

Compound or Element	Typical Analysis, as received ^(a)	Target Analysis, product, ppb
SiCl_4	98 % (min)	balance
$(\text{CH}_3)_2\text{SiCl}_2$	~2 % [max ^(b)]	---
HCl_3	0.X %	---
B	3 - 10 ppb	1
P	0.5 - 5 ppb	2
As	8.2 - 1.5 ppb	<1
Fe	40 - 100 ppb	<1
Al	40 ppb	<1
Cu	1 - 5 ppb	<1
Ni	3 ppb	<1
Mn	1 ppb	---
Pb	ND ppb	---
Sb	ND ppb	---
Sn	ND ppb	---
Ti	ND ppb	---
V	ND ppb	---

(a) Grade Al60, Union Carbide Company, Sistersville, West Virginia.

(b) Controllable to lower levels by avoiding cross product contamination in loading and shipping.



NOTES:

1. OPERATING CONDITIONS ARE FLAGGED AS FOLLOWS

PREISLISTE vom 1.1. 1981

TEMPERATURE °C

FLOW  KILograms Per Hour

2. STREAM FACTOR FOR THIS PROCESS FLOW DIAGRAM 80.7%

3. COLUMNS, REBOILERS, CONDENSERS, AND PUMPS TO BE SIZED FOR VAPOR LOADS FROM 10% TO 140% OF THOSE SHOWN.

9. READ PREREQUISITES TO BE SURED FOR UP TO DOUBLE THE FIRM SNOOWING

5. RED PUMP MUST BE LOCATED REMOTE FROM OTHER EQUIPMENT
CONNECTIONS LINES AND RED PUMP TO BE BY OTHERS).

6. OTHER CHLOROPHYLLS IN FEED MAY BE AS HIGH AS 27%
 FEED IN FEED MAY BE AS HIGH AS 1.0000

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TABLE 2. SiCl_4 PURIFICATION (C3) DESIGN
ITEMS RECEIVED FROM PAGE*

- (1) Process Flow Diagram
- (2) Mechanical Flow Diagram -- Light Ends Column
- (3) Mechanical Flow Diagram -- Heavy Ends Column
- (4) Plan Below Elevation 10'0" -- Column A
- (5) Plan Below Elevation 10'0" -- Column B
- (6) Plan Above Elevation 10'0" -- Column A
- (7) Plan Above Elevation 10'0" -- Column B
- (8) One-Line Diagram and Electrical Equipment Plan
- (9) Column Design Calculation
- (10) Panel Design
- (11) Instrumentation List
- (12) Preliminary Operating Procedure Write-Up

* This list includes those items transmitted during the visit to Houston, Texas, of January 11, 1978, (after close of report period), as most of them had been essentially completed during the report period.

Zinc Vaporizer

In the Eighth Quarterly Report, several design options for the zinc vaporizer, A7, were discussed and it was concluded that the concept of using a zinc flash vaporizer (negligible hysteresis in vapor output response with a change in metered liquid input) would be difficult if not impossible to obtain. Several factors enter into this situation.

- (1) The heat of vaporization of zinc is relatively high.
- (2) The ΔT that can be permitted between the boiling point of zinc and an outer stainless steel containment vessel (if used) through which the heat would be supplied, is quite limited, 75 C at best.
- (3) If the zinc is to be contained in graphite, which appears to be the best choice, the low conductivity of the graphite imposes a further barrier to the heat transfer.
- (4) Zinc does not wet graphite, nor any other material which is stable on contact with it, thus having thin (inventory limiting) layers of zinc in the vaporizer is precluded.
- (5) The surface tension of liquid zinc is high in contact with inert gas, resulting in a layer thickness of 0.5 cm or more in a residual pool on a plane surface; whether the surface tension in contact with saturated zinc vapor (as in a boiler) is sufficiently decreased to make a significant difference is not known.

One-dimensional and two-dimensional calculations were made to analyze the performance of a zinc vaporizer in which the heat is conducted from the outside edge of an infinitely long graphite tray to boiling zinc lying on the tray surface, i.e., approximating (except for radiation, which will be discussed later) the situation in an induction-heated cylinder containing a horizontal tray or trays parallel to the plane established by the horizontal tube diameter.

The one-dimensional calculation assumes that the tray temperature is uniform over its thickness. The more complex, two-dimensional calculations allowing for temperature gradient across the tray thickness is made with a computer code. Figure 7 shows the results in terms of zinc boil off rate as a function of distance from the wall toward the center line of the tray. The conditions and assumptions adopted for the results of Figure 7 are given in Table 3.

It is seen from the results of Figure 7 that the difference between the one- and two-dimensional results is greatest near the tray edge. As most of the boiling occurs toward the edge (79 percent of vaporization over the 50 percent of area adjacent to edge), the two-dimensionally calculated capacity is preferred.

Integration of the two-dimensional results of Figure 7 gives a tray capacity of $41.19 \text{ lb hr}^{-1} \text{ ft}^{-2}$. Thus, to supply the 61.57 lb hr^{-1} of zinc to a 25 MT/year fluidized-bed unit requires a 4.5-foot length of 4-inch-wide tray, which could be of a single length or divided in a stacked tray arrangement.

Radiation from an induction-heated graphite cylindrical envelope to the top tray of a two-tray stack would supply an addition 5.9 lb hr^{-1} capacity (assumed emissivities: graphite = 0.9, zinc = 0.3). Calculation of the radiative contribution to the underside of the trays would have to be made by iteration because of the temperature gradient (as opposed to the assumed uniform surface temperature of the boiling zinc). Carrying out this calculation was not thought to be justified at this time. The added contribution of underside radiation would be taken as a bonus.

An alternative to the zinc vaporizer design that has not been mentioned earlier is now being studied, that of inductively heating the zinc directly in a non-conducting container (quartz?). This arrangement has the advantage of eliminating the difficult problems of radiative and/or conductive heat transfer. It has the added advantage that, once the zinc has reached its boiling point, additional energy increments go entirely into vaporization as long as the zinc level (and associated radiative/conductive loss) is constant. Because of the high heat of vaporization, the response

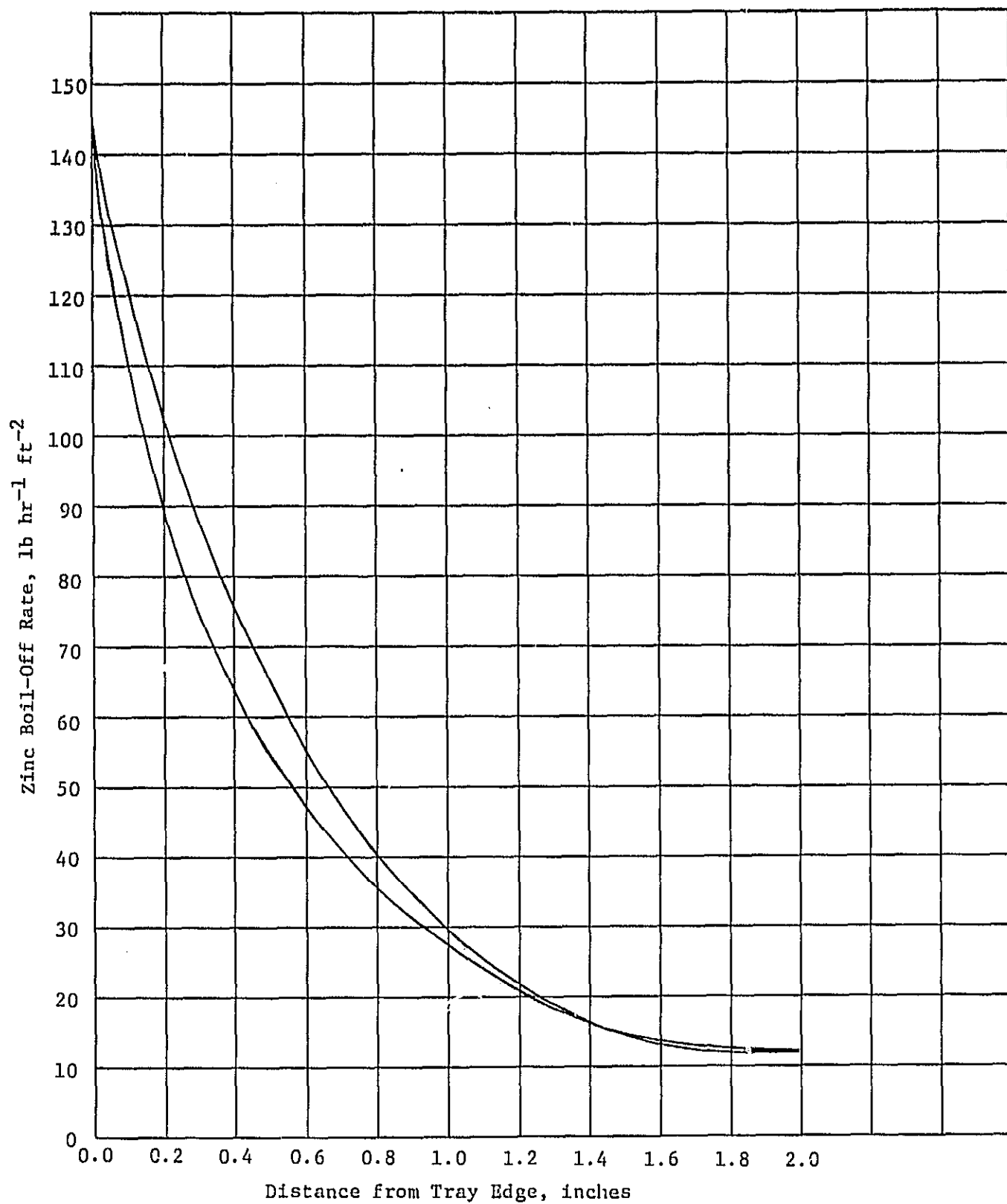


FIGURE 7. RATE OF ZINC BOILING FROM EDGE-HEATED GRAPHITE TRAY (See Table 3 for Conditions and Assumptions)

TABLE 3. CONDITIONS AND ASSUMPTIONS FOR
ZINC VAPORIZER CALCULATIONS
LEADING TO FIGURE 7 RESULTS

Tray width	=	4 inches
Tray thickness	=	0.5 inch
Thermal conductivity of graphite	=	30 BTU hr ⁻¹ ft ⁻¹ F ⁻¹
Heat transfer coefficient to boiling zinc	=	450 BTU hr ⁻¹ ft ⁻² F ⁻¹ *
Tray edge temperature	=	1967 F
Boiling point of zinc (at 1.2 atm)	=	1697 F
Zinc vapor yield	=	1.18 x 10 ⁻³ lb BTU ⁻¹

Two-dimensional calculations by BCL Computer Code XYHEAT
(Dr. C. Y. Liu)

No credit taken for heat conductivity from the wall through
the zinc itself.

* Experimentally determined; see Eighth Quarterly Report.

should be very sensitive, possibly being significantly forgiving of level fluctuations. It is too early to define the potential of this option, but experimental and theoretical evaluations are scheduled for January of 1978.

Fluidized-Bed Unit

After consideration of several fluidized-bed reactor concepts which included inputs from supportive evaluation experiments relative to selected features of each concept, a design was adopted which included some of the features of the designs shown in Figure 3 of the Eighth Quarterly Report.

The reactor is envisioned to have an outer jacket of 310 SS made from a ~9-foot section of 8-inch pipe topped with a ~3-foot expanded section of 12-inch pipe. This shell would have a ~3.5-foot graphite-lined SiCl_4 preheat section below the distributor plate, a ~6-inch distributor plate (SiC-coated graphite or sintered SiC), a reaction chamber of ~5-foot length, and an expanded section above the distributor plate. Both of the sections above the distributor would be lined with SiC-coated graphite or sintered SiC. The unit would be heated by three split electric furnaces -- one located below the distributor for preheating the SiCl_4 and the other two for the reaction and expanded sections of the unit. All furnaces would be provided with individually controlled multiple heating zones to accommodate the different heat loads required by preheating and graded-bed-temperature operation.

As is apparent from the above, plans are to introduce SiCl_4 vapor into the bottom section of the reactor where it is to be heated to 920 C before entering a ring of entry ports in the distributor plate positioned roughly midway between the center of the distributor plate and the reactor walls. Zinc vapor is introduced directly into a manifold in the distributor plate which feeds a ring of inlet ports positioned roughly midway between the SiCl_4 ports and the center of the distributor plate. Silicon seed particles are introduced into the system intermittently through the top of the system. By-products are removed from the reactor at the top of the expanded section, and the silicon product is withdrawn from the center of the distributor plate.

Temperatures of the various sections of the preheater/reactor will be monitored and controlled by thermocouples attached to the reactor wall. Bed temperatures will be monitored optically through a sight port in the top of the system. Current plans are to control the fluidized-bed height by the measured pressure differential between the bottom end of the product withdrawal tube and above the bed. This method of bed level monitoring is frequently used in the petroleum industry for large fluidized-bed systems in which fluidization is maintained at essentially a minimum level; however, it has yet to be determined whether or not it will be suitable for the highly agitated bed required for the silicon system of interest to this program. If necessary, a radioactive bed-level-monitoring system could be considered. However, such a system is less desirable because of the radiation hazard and higher cost.

Electrolytic Cell

Owing to the problems encountered with corrosion of the hot-wall containment vessel of the experimental ZnCl_2 electrolysis cell, B2, discussed in the Experimental Support section of this report, the decision has been made to abandon the hot-wall construction and adopt the more awkward but probably more reliable cold-wall (ambient) construction used by the Bureau of Mines, Reno Station. Although enclosing the insulation in the process stream would normally be undesirable, it is believed that contamination from the insulation can be avoided since the chlorine by-product will not be recycled in the 50 MT/year facility, and in a larger facility its recycle would take it through purification (as SiCl_4). As for the zinc, it is difficult to devise a mechanism by which deleterious contaminants might enter the zinc and be recycled. Of the contaminants of interest, only elemental cadmium has a vapor pressure sufficiently high to be an appreciable component of the vapor leaving the vaporizer in any case.

Accordingly, the electrolytic cell design will be drawn as scaled up to 5000 amp from the 1500-amp Bureau of Mines unit; however, greater precautions will be taken in the containment provisions to exclude external

air and avoid consequent formation of ZnO scum. In the Bureau of Mines work, economy of design was a major criterion, leading to simplified construction. In the 50 MT/year experimental facility, trouble-free operation of the less critical equipment items is a compelling objective. The degree to which containment complexity can be relaxed and economies made at that point should be discernable from subsequent operation of this type of cell both at BCL and the Bureau of Mines.

Plans for Next Report Period

Design of the 50 MT/year experimental facility is scheduled for conclusion during the next report period. Estimates will be made of the equipment, installation, and operating costs of the experimental facility and the results will be used as the basis of a refined estimate for production of semiconductor-grade silicon in a 1000 MT/year facility.

EXPERIMENTAL SUPPORT PROGRAM

During the period covered by this report, work on the experimental support program was concentrated in the following areas, to be discussed in turn.

- (1) Preparation in the miniplant of quality-evaluation material for shipment to JPL.
- (2) Evaluation of zinc displacement pump design.
- (3) Evaluation of alternative zinc vaporizer design.
- (4) Evaluation of ZnCl_2 electrolytic cell.
- (5) Evaluation of fluidized-bed design via operation of 7-inch-diameter mock-up.
- (6) Compatibility tests of materials of construction.

Preparation of Material for Evaluation

Several runs were made in the miniplant during this report period to supply JPL with quality-evaluation material. For these runs, crushed and leached ($\text{HF} + \text{H}_2\text{SO}_4$) semiconductor-grade silicon was used as the seed material, together with epitaxial-grade SiCl_4 and 99.9+ percent pure zinc* as feed materials. During the course of these runs, various arrangements have been employed to remove product from the reactor during the run so as to prevent loss of material from over-expansion of the bed. The problem with "in flight" product withdrawal is prevention of condensation of zinc and/or ZnCl_2 in the product outlet. This can be done, in principle, by purging the product exit line with inert gas. However, underpurging does not prevent condensation, and overpurging, with insufficient inert gas heating in the exit line, can lead to cooling of the bed in the area of the exit line. If the product exit line is placed in the area of high zinc concentration, overpurging of the exit line can lead to gas phase condensation of a zinc mist with resultant nucleation of silicon dust, and possible condensation of liquid zinc on the fluidized-bed product resulting in agglomeration. The latter condition is believed to occur primarily during surges of purge gas through the exit tube. Accordingly, a slow steady removal of product rather than removal by surges is to be preferred. Progress in that direction is being made; however, it should be noted that as the small scale of the operation aggravates the problem, larger scale operation should ameliorate the problem.

A significant observation has been made, albeit qualitatively, in the course of these runs: with the small depth of bed (occasioned by the limited amount of seed material available and the "in flight" removal of product) and with the increasing size of the bed particles as their seed content is decreased (providing less surface per unit volume for heterogeneous reaction), the gas-phase nucleation of silicon "dust" is increased.

* Belmont Metals, Inc., Brooklyn, New York.

Fortunately, with the increased bed depth of the 3.6 kg/hour (25 MT/year) units of the 50 MT/year experimental facility, the generation of gas-phase-nucleated silicon should decrease. It is as yet too early to predict the ultimate fraction of product that will be entrained in the by-product gases as dust, as opposed to being collected as a dense coating on the seed particles.

During the sequence of quality-material preparation runs, chlorination of the wall deposit between runs was carried out several times to demonstrate its feasibility. If the chlorination removal of upper wall deposit* is not done, the quartz reactor cracks on cool-down and must be replaced. During one wall-deposit chlorination experiment, the chlorination efficiency (conversion of Cl_2 to SiCl_4) was measured at 81 percent, or about that (80 percent) adopted for this operation in the process flow sheet (F1 through F4).

The history of the runs made during the report period to supply JPL with quality-evaluation material is shown in Figure 8. The 458-gram starting bed of semiconductor-grade seed used in Run No. 91 is traced through subsequent runs in which portions of the prior product are used as seed. The product of the three-run sequence 91-92-93 consisted of 77.4 percent deposited silicon, and that of the four-run sequence 91-92-94-95 consisted of 85.6 percent deposited silicon. A total of 1.56 kg was sent to JPL and half of the product of Run No. 94 was retained for use as seed in another material preparation run.

Evaluation of Zinc Displacement Pump Design

In support of the design effort, four trials have been made of a mock up of the mechanical displacement zinc pump, A6 (see Figure 6,

* In opting for the smaller bed and "in flight" product withdrawal to avoid loss of material, the overall bed efficiency is decreased and more silicon is deposited on the reactor wall above the bed. Returning to deeper beds in the miniplant, and, more importantly, moving to much deeper beds in the experimental facility is expected to decrease this problem to a level that can be managed as originally planned.

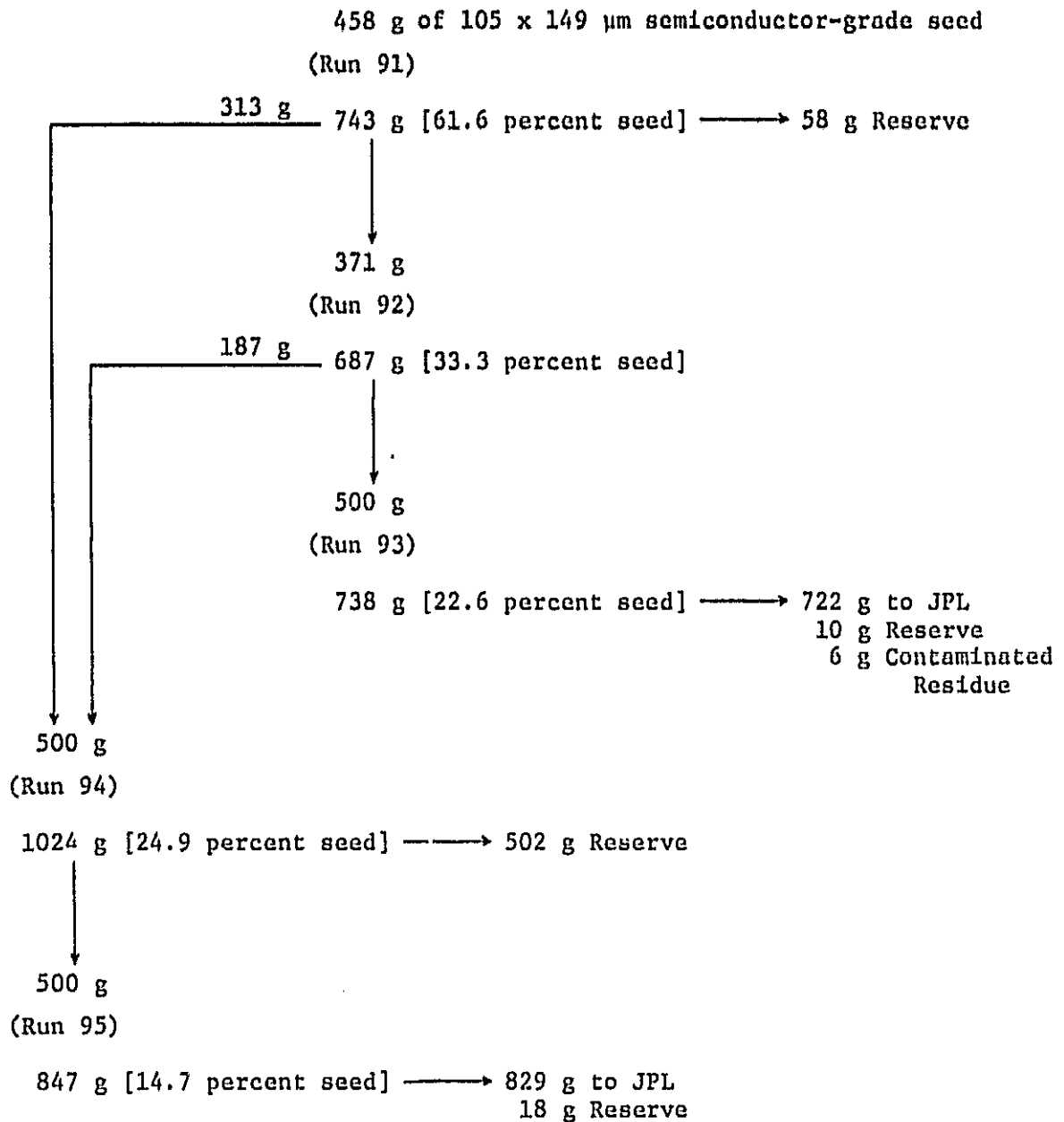


FIGURE 8. PREPARATION OF QUALITY EVALUATION MATERIAL

(The upper number in each group is the weight of the starting material, the middle number in parentheses is the run number, and the lower number is the weight of the product of that run. The bracketed number is the seed content of that product in percent. The notes to the right indicate disposition.)

Eighth Quarterly Report). In three cases, minor operational problems prevented the full evaluation desired; however, these have had nothing to do with the principle of operation. The fourth experiment demonstrated the feasibility of the design in that (1) the zinc was delivered uniformly from the feed chamber, (2) this chamber was refilled from the zinc reservoir, and (3) the feed chamber was again emptied at a controlled rate.

The major modifications to the system made during the course of this work were (1) reduction of the transfer port size to a 1/8-inch by 1/2-inch slot between the zinc reservoir and feed chamber to prevent leakage and (2) an increase in clearance between the piston and the chamber wall, except adjacent to the transfer port, to minimize binding.

Zinc Vaporizer

Two experimental runs (89 and 90/90a) were made in the miniplant during the report period to check the operation of a tray-type vaporizer (see Eighth Quarterly Report, Figure 5) having two trays with a total surface area available to zinc vaporization of about 0.12 ft², i.e., about 2 times that of the single tray 1350 C "flash vaporizer" used in most of the runs in the miniplant. The rates of vaporization obtained (e.g., 36 g/hour) with a temperature of 1100 C at the graphite shell surrounding the trays, were sufficient to support the operation of the 2-inch-diameter fluidized bed at the feed rates normally used; however, liquid zinc appeared in the quartz enclosure at higher rates. Thus, it was impossible to assess the limits of the vaporizer capacity. It was not clear whether the appearance of liquid zinc in the quartz enclosure was the result of (1) overflow (i.e., liquid feed rate exceeding vaporization capacity) or (2) condensation of zinc vapor on the quartz enclosure (which was insulated from the surrounding induction coil, but not externally heated). This problem, and the limitations in permissible heat flux through thin plates of graphite, make it difficult to carry out meaningful experiments on a small scale. The results are being analyzed as to their implications for the design of the zinc vaporizer for the experimental facility.

Plans are being made for zinc vaporization experiments in which the zinc is directly heated inductively as discussed in the Design section of this report.

Electrolytic Cell

Several Experiments were carried out during the report period in the hot-wall electrolytic cell^{*}. After the experiments reported in the Eighth Quarterly Report, the cell was refurbished to provide greater free-board in the graphite liner to avoid the problem of the foamed ZnCl_2/KCl (50/50 mole ratio) mixture from overflowing and coming in contact with the stainless steel wall and corroding it through. A subsequent electrolysis run was made, and after 23 hours of uneventful operation at 100 amps (5 amps inch^{-2}) and 5.1 volts, erratic current excursions to 150 amps were noted. After an additional 7 hours at 100 amps and 6 hours at 150 amps [voltage purposely raised to give 150 amps (7.5 amps inch^{-2})], a short developed that blew the DC power supply fuse.

Inspection of the cell on cooling revealed two problems. Some of the zinc had not coalesced, resulting in its not having drained from the cathode surface. It is not inconceivable that at operating temperature (500 C), the undrained zinc had indeed bridged the 0.5-inch gap between anode and cathode. Coalescence appears to have been prevented by a skin, presumably of zinc oxide, on the zinc surface.

Further examination of the dismantled cell revealed a salt accumulation in one section of the annulus between the graphite and the stainless steel, and partial corrosion in the area. Discoloration of the wall was observed immediately above, which is taken to be evidence that the electrolyte had foamed and had overflowed the graphite crucible at that

* The general construction of the cell is shown in Figure 20 of the Fifth/Sixth Quarterly Report. However, horizontal electrodes rather than the vertical electrodes shown have been employed, approximating the Bureau of Mines electrode design.

location, which corresponded to the side of the anode at which chlorine was evolved from the slanted gas channels on the underside.

A subsequent experiment in a quartz container showed considerable gas evolution when the ZnCl_2 and KCl were mixed at $\sim 320^\circ\text{C}$. It is believed that the gas evolution (and foaming) resulted from residual moisture in the ZnCl_2 *. The product of reaction of the moisture with the ZnCl_2 would be ZnO which could act to stabilize bubbles and permit formation of a foam during chlorine evolution. The presence of zinc oxide prevents the coalescence of zinc. Thus it is believed that most, if not all, of the problems experienced with the electrolytic cell stem from the presence of excessive moisture in the zinc chloride. It is not anticipated that such problems will be encountered with the anhydrous by-product of the experimental facility.

A supply of presumably anhydrous zinc chloride is available from operation of the miniplant**; however, further work with the electrolytic cell has been deferred, since although the hot-wall construction was convenient for fabrication of the experimental cell, it has been decided that the design of the cells for the experimental facility would be of the "cold"-wall type and follow more closely the Bureau of Mines practice.

The course of future experimental work with the electrolysis cell is currently under discussion.

Fluidized-Bed Mock-Up

A full scale (7-inch-diameter) mock-up of the 25 MT/year fluidized-bed unit was assembled so as to evaluate the bed action and gas mixing as a function of gas flow. A group of eight holes is provided around the center of the distribution plate to function as "zinc" inlets, and a group of 12 holes surrounding that provides for " SiCl_4 " introduction.

* Technical grade, anhydrous (undistilled), Eaton-Colby Chemical Company, Columbus, Ohio.

** Much of the miniplant by-product has been discarded, but 120 kg has been set aside for future experiments.

In the mock up, compressed air is used as the fluidizing gas, with the velocity of the air being based on relative density and viscosity so as to approximate the fluidization condition for zinc and SiCl_4 vapors at 900 C. In many applications of fluidized beds, modelling is complicated by the fact that the gases enter at temperatures much lower than that of the bed, and considerable gas expansion occurs in the bed as the gas is heated. However, in the case at hand, the reactants must be preheated to reaction temperature before entering the bed (to prevent zinc mist condensation). Thus, the complication of gas expansion in the bed is obviated, and modelling with air at ambient temperature reliably represents the fluidization pattern for the condition being modelled. Gas mixing in the reactor can be followed by introducing smoke through one or another of the reactant inlet groups, or HCl-containing air through one set and NH_3 -containing air through the other to form NH_4Cl smoke by reaction in situ.

In preliminary experiments with the model, the fluidization pattern appeared to be satisfactory; however, an additional inlet port had to be added to the outer " SiCl_4 " inlet manifold to obtain uniform flow distribution. Modelling experiments will be continued in the next report period with the objective of observing the effects of particle size, bed height, and relative flow through the gas distributor on fluidization and on mixing of the two gas streams. In addition, distributor plate designs will be studied.

Materials Compatibility

During the current report period, several experiments were carried out to determine the compatibility of potential materials of construction.

To determine the corrosion resistance of stainless steel to zinc chloride, a sample of 304 stainless steel was exposed to zinc chloride for 72 hours at 500 C. No corrosion of the stainless steel was indicated by appearance or weight loss. The residual zinc chloride showed no color, and spectrographic analysis gave no evidence of the presence of iron,

chromium, or nickel. Thus, it is highly probable that stainless steel can be used in contact with zinc chloride, at least to 500 C, from the standpoint of equipment durability. However, the question of possible transfer of contamination to the silicon product deserves further consideration. Although spectroscopically undetectable quantities of (e.g.) iron chloride from stainless steel corrosion would be electrolyzed and the iron go into solution in the zinc, the one-step distillation during zinc vaporization should leave the iron behind. The vapor pressure of iron is 10^{-10} atm at the boiling point of zinc, and when that factor is multiplied by the activity of iron in zinc at a low ppm level, an infinitesimal transfer results. Only in the case of entrainment of unvaporized zinc droplets from the vaporizer could significant impurity transfer result from that source.

The corrosion of stainless steel by liquid zinc is a different matter. The zinc penetrates the grain structure of stainless steel and as both iron and nickel are highly soluble in zinc, the stainless steel disintegrates. To check the transport of zinc vapor through the slight porosity of ATJ graphite, a sample of 304 stainless steel was encapsulated in an ATJ graphite "test tube" (0.5-inch wall thickness) provided with a steel wool "getter" section at the top plugged on both sides to make sure that penetration of zinc could not occur through the originally open end of the tube. After 100 hours in zinc vapor at 850 C, the stainless steel slugs at the bottom of the test tube had been almost completely infiltrated with zinc which apparently diffused through the graphite (no cracks in the graphite were observed).

It is evident from the above that any design involving graphite barriers between zinc and stainless steel must provide for elimination of the porosity in the graphite and avoidance of cracked joints, etc.

It is not planned that stainless steel will be used in contact with the ZnCl_2 -KCl mixture in the electrolytic cell, as the KCl content probably would lead to the solution of the more electrovalent protective chlorides on the surface of the stainless steel (FeCl_2 , NiCl_2) and open the surface to chlorine corrosion as suggested by the results of the

electrolytic cell experiments described above. Accordingly, no corrosion tests are contemplated of the ZnCl_2/KCl mixture versus stainless steel.

Work Planned for the Next Report Period

The following work is planned for the next report period.

- (1) Preparation of material for quality evaluation by JPL and/or its subcontractors will be continued.
- (2) Experiments will be continued with the zinc displacement pump to permit refinement of the design.
- (3) The concept of heating zinc directly by induction will be experimentally evaluated.
- (4) Experiments will be planned and executed to define the factors wherein the electrolytic recovery of zinc from the zinc/ ZnCl_2 by-product (containing some silicon dust) differs from the electrolysis of ZnCl_2 developed by the Bureau of Mines.
- (5) Experiments will be continued in the full-scale model of the fluidized bed.
- (6) Other experiments will be performed as may be needed in support of the design work.

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